IS 11015 (1984): Ammonium Chloride for Cosmetic Industry [PCD 19: Cosmetics]

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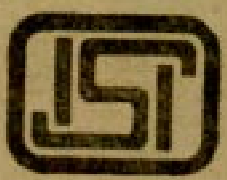
IS : 11015 - 1984

(Reaffirmed 2005)

Indian Standard

SPECIFICATION FOR AMMONIUM CHLORIDE FOR COSMETIC INDUSTRY

UDC 661·521 : 665·58



INDIAN STANDARDS INSTITUTION
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Gr 3

November 1984

Indian Standard

SPECIFICATION FOR AMMONIUM CHLORIDE FOR COSMETIC INDUSTRY

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Indian Standard

SPECIFICATION FOR AMMONIUM CHLORIDE FOR COSMETIC INDUSTRY

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 24 July 1984, after the draft finalized by the Cosmetics Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

0.2 IS : 1113-1965* covers a number of grades used in a variety of industries. The present standard has been drawn up specifically to cater to the needs of the cosmetic industry.

0.3 In keeping with the general pattern of cosmetic raw material standard specification in this standard also a single set of requirements has been stipulated to facilitate implementation. All other changes considered necessary to align the standard with others in the series for raw materials and cosmetics have also been included.

0.4 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960†. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

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1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for ammonium chloride intended for use in the cosmetic industry.

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*Specification for ammonium chloride, technical and pure (*revised*).
†Rules for rounding off numerical values (*revised*).

2. REQUIREMENTS

2.1 Description — Ammonium chloride which is somewhat hygroscopic, shall be in the form of white crystals, pellets or friable bars free from hard caking, perceptible odour and also any visible impurities.

2.2 The material shall also comply with the requirements specified in Table 1 when tested according to the methods given in Appendix A. Reference to the relevant clauses in Appendix A is given in col 4 of the table.

TABLE 1 REQUIREMENTS FOR AMMONIUM CHLORIDE FOR COSMETIC INDUSTRY

(*Clauses* 2.2, A-7.3.1, A-8.3.1 *and* B-5.3)

SL No.	CHARACTERISTIC	REQUIREMENT	METHODS OF TEST (REF TO CL NO. IN APPENDIX)
(1)	(2)	(3)	(4)
i)	Loss on drying, percent by mass, *Max*	1	A-3
ii)	Ammonium chloride as (NH_4Cl) percent by mass, (dry basis), *Min*	99·5	A-4
iii)	Total sulphated residue on sublimation, percent by mass, *Max*	0·3	A-5
iv)	Arsenic (as As_2O_3), ppm, *Max*	5	A-6
v)	Heavy metals (as Pb), ppm, *Max*	10	A-7
vi)	Iron (as Fe), percent by mass, *Max*	0·01	A-8

3. PACKING AND MARKING

3.1 The material shall be packed in multi-walled paper or jute bags lined with polyethylene films (*see* IS : 2508-1977*) or in such other containers as agreed to between the purchaser and the supplier.

3.2 The containers shall be securely closed and marked with the following information:

a) Name of the materials;

b) Name of the manufacturer;

c) Gross and net mass of the material in the container;

d) Recognized trade-mark, if any; and

e) Month and year of manufacture.

*Specification for low density polyethylene films.

3.2.1 The containers may also be marked with the ISI Certification Mark.

NOTE — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act and the Rules and Regulations made thereunder. The ISI Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions, under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

4. SAMPLING

4.1 The method of drawing representative samples of the material and the criteria for conformity shall be as prescribed in Appendix B.

APPENDIX A

(*Clause* 2.2)

A-1. QUALITY OF REAGENTS

A-1.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1977*), shall be employed in tests.

NOTE — ' Pure chemicals ' shall mean chemicals that do not contain impurities which affect the results of analysis.

A-2. PREPARED SAMPLE

A-2.1 Mix the sample well by shaking the bottle several times and transfer a portion immediately to a wide-mouth bottle and stopper it. Take care that no pieces of cork or sealing wax get mixed with the material. Do not expose the prepared sample to an atmosphere containing acid or alkali fumes.

A-3. DETERMINATION OF LOSS ON DRYING

A-3.1 Procedure — Weigh accurately about 5 g of the prepared sample in a weighed shallow porcelain dish and dry in an air oven at 100 to 105°C for two hours to constant mass.

*Specification for water for general laboratory use (*second revision*).

A-3.2 Calculation

$$\text{Loss on drying, percent by mass} = \frac{100 \times M_1}{M}$$

where

M_1 = loss in mass in g on drying, and
M = mass in g of the material taken for test.

A-4. DETERMINATION OF AMMONIUM CHLORIDE

A-4.1 Reagents

A-4.1.1 *Nitric Acid* — Concentrated nitric acid, diluted with an equal volume of water.

A-4.1.2 *Potassium Chloride* — Analytical reagent grade. Heat to constant mass at 500°C. Equivalent weight 74·555.

A-4.1.3 *Potassium Chromate Solution* — 5 percent solution in water (*m*/*m*).

A-4.1.4 *Standard Silver Nitrate Solution* — approximately 0·1 N. Keep in amber coloured stoppered bottle away from light.

A-4.1.4.1 *Standardization* — Weigh accurately enough potassium chloride to yield titration volume of about 40 ml (about 0·3 g for 0·1 N solution) to a 250-ml conical flask and dissolve in 40 ml of water. Add 1 ml of potassium chromate solution and titration with silver nitrate solution until first perceptible pale brown coloration appears. Do a blank to determine the volume of silver nitrate solution required to produce end point colour in 75 ml of water containing 1 ml of potassium chromate solution. Subtract the blank reading from the titration volume. From the net volume of silver nitrate calculate normality as follows:

Calculation

$$\text{Normality, } N = \frac{M \times 1\,000}{V \times 74{\cdot}555}$$

where

M = mass of potassium chloride in g, and
V = volume of silver nitrate in ml.

A-4.1.5 *Ferric Alum Indicator* — A saturated solution of ferric ammonium sulphate [$Fe\,NH_4\,(SO_4)_2 \cdot 12\,H_2O$] in water.

A-4.1.6 *Potassium or Ammonium Thiocyanate Standard Solution* — Prepare approximately 0·1 N from potassium or ammonium thiocyanate which is free from chloride. Determine working titre as follows.

A-4.1.6.1 Accurately measure 50 ml of standard silver nitrate solution into a conical flask. Add 2 ml of ferric alum solution and 5 ml of nitric acid and titrate with thiocyanate solution until solution appears pale rose after vigorous shaking.

A-4.2 Procedure

A-4.2.1 Weigh accurately about 0·2 g of the dried sample (dried in accordance with **A-3.1**) and dissolve in 40 ml water in a conical flask. Add 3 ml nitric acid and 5 ml of nitrobenzene. Add exactly 50 ml of the standard silver nitrate solution and shake vigorously for one minute. Add 2 ml of ferric ammonium sulphate solution and titrate with standard thiocyanate solution until the solution appears pale rose after vigorous shaking. Determine the volume of silver nitrate consumed by the sample.

A-4.2.2 Calculation

$$\text{Ammonium chloride, percent by mass (on dry basis)} = \frac{0{\cdot}5349 \times V \times N}{M_1}$$

where

V = volume in ml of standard silver nitrate solution consumed by sample,

N = normality of standard silver nitrate solution, and

M_1 = mass in g of dry sample taken.

A-5. DETERMINATION OF TOTAL SULPHATED RESIDUE ON SUBLIMATION

A-5.1 Reagent

A-5.1.1 *Concentrated Sulphuric Acid* — conforming to IS : 266-1977*.

A-5.2 Procedure — Weigh accurately about 5 g of the dried material (**A-3.2.1**) into a dry weighed crucible, heat until most of the material sublimes off. Add about 1 ml of concentrated sulphuric acid. Mix with the residue and finally ignite to constant mass.

*Specification for sulphuric acid (*second revision*).

A-5.3 Calculation

Total sulphated residue after sublimation, percent by mass $= 100 \times \frac{M_1}{M_2}$

where

M_1 = mass in g of the residue, and

M_2 = mass in g of the dried material taken for the test.

A-6. TEST FOR ARSENIC (as As_2O_3)

A-6.1 Procedure — Dissolve 1·0 g of the material in 10 ml of water. Carry out test for arsenic as prescribed in IS : 2088-1971*, using for comparison a stain obtained with 0·005 mg of arsenic trioxide.

A-6.1.1 The material shall be taken to have satisfied the requirement of the test if the length and intensity of the stain is not greater than that produced in the control test.

A-7. TEST FOR HEAVY METALS

A-7.1 Apparatus — Nessler cylinders — 50 ml capacity.

A-7.2 Reagents

A-7.2.1 *Acetic Acid* — 1 N.

A-7.2.2 *Sodium Sulphide Solution* — Dissolve 10 g of sodium sulphide in water and make up to 100 ml. Filter and preserve in a dark coloured bottle.

A-7.2.3 *Standard Lead Solution* — Dissolve 1·83 g of reagent grade lead acetate [$Pb(C_2H_3O_2)_2.3H_2O$] in 100 ml of water and add a few drops of acetic acid to clear any cloudiness. Dilute the solution to 1 000 ml. Take 10 ml of this solution and make it into 1 000 ml. One millilitre of this solution contains 0·01 mg of lead (as Pb).

A-7.3 Procedure — Dissolve 1·0 g of material in 2 ml of acetic acid in a Nessler cylinder. Add 20 ml of water and then 10 ml of sodium sulphide solution. Make up the volume to 50 ml. Carry out a control test in another Nessler cylinder using 1 ml of standard lead solution in place of the material and 2 ml of acetic acid and 10 ml of sodium sulphide solution. Make the total volume same as earlier. Shake the two Nessler cylinders well. Compare the intensity of colour produced in the two cylinders.

*Methods for determination of arsenic (*first revision*).

A-7.3.1 The limit prescribed in Table 1 shall be considered as not having been exceeded if the colour produced by the material is not greater than that obtained in the control test.

A-8. TEST FOR IRON

A-8.1 Apparatus

A-8.1.1 *Nessler Cylinders* — 50 ml capacity.

A-8.2 Reagents

A-8.2.1 *Hydrochlric Acid* — *See* IS : 265-1976*.

A-8.2.2 *Concentrated Sulphuric Acid* — *See* IS : 266-1977†.

A-8.2.3 *Ammonium Persulphate* — Solid.

A-8.2.4 *Butanolic Potassium Thiocyanate Solution* — Dissolve 10 g of potassium thiocyanate in 10 ml of water and make up the solution to 100 ml with *n*-butanol. Shake vigorously until the solution is clear.

A-8.2.5 *Standard Iron Solution* — Dissolve 0·702 g of ferrous ammonium sulphate [$Fe\,SO_4 \cdot (NH_4)_2SO_4 \cdot 6H_2O$] in water in a one litre flask. Add 10 ml of concentrated sulphuric acid and dilute with water to the mark. When required for test, dilute 10 ml of this solution to 100 ml in a volumetric flask. One millilitre of this diluted solution contains 0·01 mg of iron (as Fe).

A-8.3 Procedure — Dissolve 1·00 g of the material in hot water. Cool and transfer to a Nessler cylinder. Add 2 ml of hydrochloric acid, 30 mg of ammonium persulphate and 15 ml of butanolic potassium thiocyanate solution. Make up to 50 ml with water, shake vigorously for 30 seconds and allow the liquids to separate. Carry out a control test in another Nessler cylinder with 10 ml of standard iron solution in place of the sample and the same quantities of other reagents in the same total volume of the reaction mixture. Compare the colour of the butanol layers in the two cylinders.

A-8.3.1 The limit prescribed in Table 1 shall be taken as not having been exceeded if the intensity of the colour of the butanol layer in the test with the material is not more than that produced in the control test.

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*Specification for hydrochloric acid (*second revision*).
†Specification for sulphuric acid (*second revision*).

APPENDIX B

(*Clause* 4.1)

SAMPLING OF AMMONIUM CHLORIDE FOR COSMETIC INDUSTRY

B-1. GENERAL REQUIREMENTS OF SAMPLING

B-1.0 In drawing, preparing, storing and handling test samples, the precautions and directions as directed in IS : 8883 (Part 1)-1978* shall be followed.

B-2. SCALE OF SAMPLING

B-2.1 Lot — All the containers in a single consignment of the material of the same grade and type, drawn from a single batch of manufacture, shall constitute a lot. If a consignment is declared to consist of different batches of manufacture, the batches shall be marked separately and the group of containers in each batch shall constitute separate lots. In the case of a consignment drawn from a continuous process, 1 000 containers (or 100 tonnes) of the material shall constitute a lot.

B-2.2 The number of containers to be chosen from a lot shall depend on the size of the lot and shall be as given in Table 2.

TABLE 2 NUMBER OF CONTAINERS TO BE SELECTED FOR SAMPLING

LOT SIZE (N)	NUMBER OF CONTAINERS TO BE SELECTED (n)
(1)	(2)
Up to 50	3
51 to 150	4
151 to 300	5
301 to 500	7
501 and above	10

B-2.3 These containers shall be chosen at random from the lot, and in order to ensure randomness of selection a random number table as agreed to between the purchaser and the supplier shall be used. In case

*Methods of sampling chemicals and chemical products: Part 1 General requirements and precautions.

such a table is not available, the following procedure may be adopted:

Arrange all the containers in the lot in a systematic manner and starting from any container, count them as 1, 2, 3, ... up to r and so on, r being equal to the integral part of N/n. Every *rth* container thus counted shall be withdrawn and all such containers shall constitute the sample.

B-3. TEST SAMPLES AND REFEREE SAMPLE

B-3.1 Draw with an appropriate sampling instrument small portions of the material from different parts of each container selected (*see* Table 2). The total quantity taken out from each container shall be sufficient to conduct the tests for all the characteristics given in **2**.

B-3.2 Mix thoroughly all portions of the material drawn from the same container to form an individual test sample. Out of these portions equal quantities from all individual test samples so formed shall be mixed together to form a composite test sample.

B-3.3 All the individual test samples and the composite test sample shall be divided into three equal parts, thus forming three sets of test samples. These parts shall be immediately transferred to thoroughly dried bottles which shall then be sealed air-tight with glass stoppers. These shall be labelled with all the particulars of sampling given under **B-1.7**. One of these sets of test samples shall be sent to the purchaser and another to the supplier.

B-3.4 Referee Sample — The third set of test samples, bearing the seals of the purchaser and these supplier, shall constitute the referee sample and shall be used in case of dispute between the purchaser and the supplier. It shall be kept at a place agreed to between the purchaser and the supplier.

B-4. NUMBER OF TESTS

B-4.1 Test for the determination of ammonium chloride shall be conducted on each of the individual test samples.

B-4.2 Tests for the remaining characteristics given in **2** shall be conducted on the composite tests samples.

B-5. CRITERIA FOR CONFORMITY

B-5.1 For Individual Samples — From the test results for ammonium chloride, the mean (X) and range (R) shall be calculated (range being defined as the difference between the maximum and the minimum of the test results).

B-5.2 The lot shall be declared as conforming to the requirement of ammonium chloride if the value of the expression ($X - 0{\cdot}6\,R$) is greater than or equal to 99·5.

B-5.3 For Composite Sample — For declaring the conformity of the lot to the requirements of all other characteristics tested on the composite sample, the test result for each of the characteristics shall satisfy the relevant requirement specified in Table 1.